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Figure 2h

Supporting Information for Szyperski *et al.* (2002) *Proc. Natl. Acad. Sci. USA*
99 (12), 8009–8014. (10.1073/pnas.122224599).

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Supporting Figure 9

Fig. 9. Experimental scheme for the 3D HCCH-COSY experiment. Rectangular 90° and 180° pulses are indicated by thin and thick vertical bars, respectively, and phases are indicated above the pulses. Where no rf phase is marked, the pulse is applied along x . The scaling factor k for ^1H chemical shift evolution during t_1 is set to 1.0. The high power 90° pulse lengths were: 5.8 ms for ^1H and 15.4 ms for ^{13}C , and 38 ms for ^{15}N . The lengths of the ^1H spin-lock purge pulses are: first SL_x , 2.8 ms; second SL_x , 1.7 ms; SL_y , 4.9 ms. SEDUCE is used for decoupling of ^{13}CO during t_1 and t_2 (rf field strength = 1 kHz). The WURST scheme is used for decoupling of ^{13}C during acquisition. The ^1H carrier is placed at the position of the solvent line at 0 ppm before the start of the first semiconstant-time ^1H evolution period, and then switched to the water line at 4.78 ppm after the second 90° ^1H pulse. The ^{13}C and ^{15}N rf carriers are set to 38 ppm and 120.9 ppm, respectively. The duration and strengths of the pulsed z-field gradients (PFGs) are: G1 (500 ms, 6 G/cm); G2 (500 ms, 7 G/cm); G3 (100 ms, 12 G/cm); G4 (100 ms, 12.5 G/cm); G5 (2 ms, 9 G/cm); G6 (500 ms, 5 G/cm); G7 (1.5 ms, 8 G/cm); G8 (400 ms, 6 G/cm). All gradients are applied along z axis and are of rectangular shape. All PFG pulses are of rectangular shape. A recovery delay of at least 100-ms duration is inserted between a PFG pulse and an rf pulse. The delays are: $t_1 = 1.6$ ms, $t_2 = 850$ ms, $t_3 = 2.65$ ms, $t_4 = 3.5$ ms, $t_5 = 7$ ms, $t_6 = 1.6$ ms, $t_7 = 3.2$ ms. Phase cycling: $f_1 = x$; $f_2 = x, -x$; $f_3 = x, -x$; $f_4 = x$; $f_5(\text{receiver}) = x, -x$. Quadrature detection in $t_1(^{13}\text{C}/^1\text{H})$ and $t_2(^{13}\text{C})$ is accomplished by altering the phases f_2 and f_3 , respectively, according to States-TPPI. Water suppression is accomplished by coherence pathway rejection using spin-lock purge pulses and pulsed field z -gradients. For acquisition of central peaks derived from ^{13}C steady state magnetization, a second data set with $f_1 = -x$ is collected. The sum and the difference of the two resulting data sets generate subspectra II and I, respectively, containing the central peaks and peak pairs.

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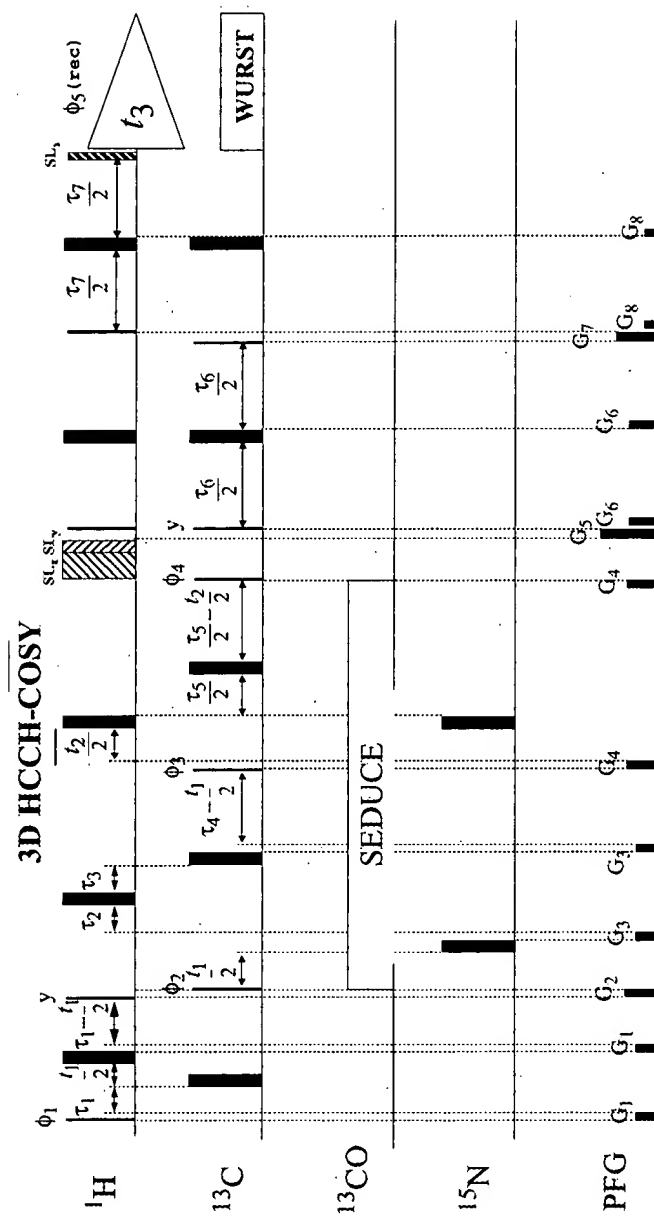


Figure 9